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LASER SPOT SCANNING OF PHOTOELECTROCHEMICAL CELLS.(U)

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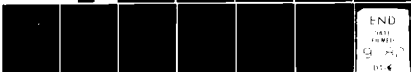
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LASER SPOT SCANNING OF PHOTOELECTROCHEMICAL CELLS

by

M. E. Langmuir, R. H. Micheels, R. A. Boudreau and R. D. Rauh

Presented at

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Measurement Techniques for Photoelectrochemical Solar Cells  
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## LASER SPOT SCANNING OF PHOTOELECTROCHEMICAL CELLS

M. E. Langmuir, R. H. Michaels, R. A. Boudreau and R. D. Rauh  
EIC Laboratories, Inc., 111 Chapel Street, Newton, MA 02158

### ABSTRACT

Laser scanning of the photoelectrode in a photoelectrochemical cell is a powerful diagnostic tool for assessing the effect of surface modifications of the semiconductor electrode on cell performance. Photocurrent scan maps of photoelectrodes with external and/or white light bias allow the study of the effect of iR losses in the cell under operating conditions. A dye laser adds versatility to the system by permitting wavelength dependent responses to be mapped. The possibility of time resolved measurements, using a pulsed laser source, is also discussed.

### INTRODUCTION

The method, which involves the recording of photocurrent or photovoltage developed in a semiconductor as a spot of light is raster scanned over the semiconductor surface, has been referred to in the literature by a variety of descriptive names including scanning light spot analysis (SLS) (1), scanning light microscopy (SLM) (2) and simply, laser scanning (3). The scanning has been accomplished by translating the laser beam in both x and y directions with oscillating mirrors (2,3) or by translating the stage on which the semiconductor or solar cell is mounted (1). The scanner which we describe here is of the former type. By simply changing the position and focal length of the focusing lens, resolution may be varied to study single grain boundaries in polycrystalline semiconductors, or to map losses in large area solar cells. The use of laser scanning to study grain boundaries has been described elsewhere (2,4) and will not be treated here. In this paper, examples are given of the utility of the method for the study of 1) surface modifications of a thin film CdSe photoelectrode and 2) losses in photoelectrochemical cells (PECs) which can be ascribed to cell design.

### INSTRUMENTATION

The laser spot scanner system, pictured in Figure 1, is based on two galvanometer scanners (General Scanning G100-PD) and a 3.5 mW HeNe laser source attenuated with neutral density filters and focused with a plano-convex lens. Lenses of varying focal lengths were used for different resolution and scan range requirements. For high resolution studies, a 50 mm focal length lens,  $L_1$ , placed after the scanner mirrors, was used which produced a spot with an  $11\mu$  (1/e) diameter. For large cells a 200 mm lens,  $L_2$ , placed before the scanner mirror,

was used instead. The scanner mirrors were driven in a raster pattern by two ramp generators through two scan control amplifiers (General Scanning CCX-101-1). Photocurrent was amplified and monitored with a potentiostat (Amel 551) for fast scan rates and a picoammeter (Keithly 480) for slow scan speeds. Photovoltage was monitored with a 100 kHz bandwidth amplifier. The two-dimensional raster scanned photocurrent and photovoltage images were either displayed on an oscilloscope, used in the x-y mode, for rapid scanning, or on an x-y chart recorder for slow scans. The oscilloscope images were produced by modulating the CRT beam intensity with the photocurrent or photovoltage signal. When the x-y recorder was employed, a "X-modulation" scheme was used in which the photocurrent or photovoltage signal is superimposed on the X-position input to the recorder with an op-amp adder, allowing quantitative measurements to be made. A pen lifting circuit was also used with the chart recorder to blank the retrace in the Y-scan direction.

In order to obtain additional information from spot scanning, a  $N_2$  pumped, pulsed dye laser (Molelectron DL II) was employed as a scanner light source. The laser wavelength was tuneable over the range 360-940 nm and the pulse width was about 5 nsec. The pulsed photovoltage signal was averaged with either a boxcar integrator or a lock-in amplifier depending on the signal decay time.

#### STUDY OF SURFACE MODIFICATIONS OF THE PHOTOELECTRODE

The effect of surface modifications of electrodeposited CdSe on Ti foil are shown in Figure 2. A  $1\text{ cm}^2$ , 4 micron thick film was subjected to the following treatments in succession: A, annealing at  $600^\circ\text{C}$  in air for 15 minutes; B, a 2-second etch with conc. HCl; C, anodization for 3 seconds in  $0.1\text{N H}_2\text{SO}_4$  at 4.5V. The PEC parameters after each treatment were measured in aqueous polysulfide electrolyte at  $45\text{ mW/cm}^2$  intensity with an IR filtered tungsten-halogen lamp (see Table 1). Figure 2 shows the corresponding laser scan maps and a photograph of the electrode surface after treatment C.

The annealed electrode had a low photoresponse with a slow time constant. After the HCl etch, the response was rapid and almost a factor of 10 higher, although some bare spots appeared as indicated by the small dips in the photoresponse. The drastic reduction of the PEC parameters after anodization were attributable to cracking and flaking off of the film as shown by a comparison of the scan map and the corresponding photograph of the electrode surface.

Often, the changes in photoresponse with surface modification are subtle and the measurements of PEC parameters are very sensitive to experimental conditions such as light intensity at the photoelectrode and reproducible positioning. If several surface modifications can be made on one electrode and recorded on one scan map, this difficulty is obviated. Figure 3 is an example of an array of etch "steps" on a single chemical bath deposited CdSe film on titanium. Virtually no

difference was observed at short circuit, but when the cell was held at  $-0.4V$ , i.e., close to the open circuit voltage, the etch steps could be discerned. Thus, changes in collection efficiency with surface modification should be measured at small band bending. The use of forward bias has also been shown to be advantageous to detect defects in solid state photocells (3).

Much interest has been shown recently in the  $ZnCl_2$  treatment of CdSe and CdS photoelectrodes (5-8). The treatment apparently suppresses the opposing cathodic current at the photoelectrode and thereby enhances the photocurrent and the open circuit photovoltage. To investigate whether it had any effect on subgap states usually present on polished CdSe crystal surfaces, a  $1\text{ cm}^2$  single crystal was given the localized combination of surface treatments shown in Figure 4A. At a bias voltage of  $-0.45V$  vs. a CoS counter electrode in aqueous polysulfide electrolyte, the photocurrent on the polished half of the electrode was very small compared to that on the etched half. Figure 4B shows the scan map of the etched portion of the crystal. An increase in photocurrent of  $\sim 10\%$  is observed when the beam crosses into the region of  $ZnCl_2$  treatment (indicated by an arrow in Figure 4B).

To determine whether  $ZnCl_2$  had any effect on subgap states, the HeNe CW laser was replaced with a  $N_2$ -pumped dye laser and a lock-in amplifier. The normalized action spectra of a polished and etched portion of the crystal are shown in Figure 5A. The response for the polished area is at least a factor of 10 smaller than that of the etched area and shows a subband gap absorption maximum at about 760 nm in agreement with similar spectra reported by Heller et al. (9). Using 760 nm pulsed light from the dye laser we recorded two single photovoltage scans to cover each of the 4 quadrants of the crystal and repeated the scans in the same positions with 588 nm pulsed light. The average power at the two wavelengths was approximately 20  $\mu W$  at 760 nm and 100  $\mu W$  at 588 nm, and the beam diameter was about 50  $\mu m$  in these experiments. The scans at 760 nm do indicate that the subband gap response is decreased by etching and the 588 nm response is increased by etching. The  $ZnCl_2$  also appears to diminish slightly the subband gap response in both polished and etched regions.

To further characterize the different regions of the crystal, the time resolved rise and decay of the photovoltage signals caused by band gap and subband gap excitation were recorded on an oscilloscope triggered by the laser pulse. The subband gap excited photovoltage rise and decay times are not affected by etching but the rise time of the band gap response is much faster in the etched than in the polished area. In principle, it should also be possible to map the fast and slow response areas using a boxcar integrator to record the signal.

Although we cannot yet interpret with any certainty the observed spectral and kinetic differences in photovoltage response, the

sensitivity to surface treatment indicates the participation of charge traps, at or near the solid-liquid interface, which act as recombination sites for holes and electrons. Excitation of charge from these traps to the conduction band, for example, could result in the well-defined subband gap response. The different mechanisms of filling and emptying these traps under sub- and supraband gap excitation may explain the observed differences in photovoltage transients.

#### STUDY OF CELL DESIGN

With additional modification of the basic scanner to provide for white light illumination of the photoelectrode while scanning with the chopped HeNe beam, it was possible to detect areas of iR drop in PECs as well as surface defects in the photoelectrode. This method has been used to analyze two possible practical frontwall cell formats for thin film CdSe electrodes shown in Figure 7.

Cell I is a structure with holes in the planar photoelectrode, to enable ion flow to the rear-placed counter electrode. The photoelectrode is of  $\sim 15 \text{ cm}^2$  nominal area, produced by chemical bath deposition of CdSe on Ti foil. The scan map of a portion of this cell, shown in Figure 9, was obtained using a secondary white light bias to enhance iR effects. In addition to revealing the dead areas resulting from the perforations, the map shows pinholes and several areas of enhanced photocurrent scratches in the substrate, where the film is perhaps thicker or of greater roughness. Cell polarization and iR effects show up as slightly enhanced photocurrents immediately adjacent to the holes, and as a general negative curvature of the scans in areas between the holes.

Cell II contains a  $100 \text{ cm}^2$  CdSe photoelectrode, prepared by electrodeposition on a louvered Ti substrate. In this case, the slots beneath the louvers provide the path for ion flow. Figure 8 shows a scan map of the illuminated portion of the electrode scanned vertically across 3 of the louvers. At the right side of Figure 8 is a cross-sectional view of the louvers. Maximum photocurrent occurs where the distance between the photoelectrode and the counter electrode is a minimum, i.e., at the flat edge. Areas of maximum loss are, of course, the slits between the louvers, indicated by A. This suggests that 1) the louvers should be narrower to produce more edges, 2) the angle of bend should be smaller, and 3) a different design in which the louvers overlap slightly would decrease the loss area. Other areas of loss marked B and C are due to bare spots on the Ti substrate.

#### CONCLUSION

We have demonstrated that laser spot scanning is a useful tool for studying the preparation of surfaces of semiconductors employed in photoelectrochemical cells. Spot scanning is also an aid in designing practical geometries for such devices. Clearly, however, the spot



scanning technique using liquid-semiconductor junctions goes beyond the parochial concerns of solar cell studies. The technique can be employed in the rapid evaluation of bulk and surface properties of semiconductors without the need of prior processing. The potential exists for interpreting localized responses to light in terms of diffusion length, surface recombination velocity, minority carrier lifetime and surface state density, for example, so that surface profile of these properties may be constructed on as-grown semiconductor materials. Hence, this measurement technique can be used to evaluate semiconductors for a variety of device applications where lateral homogeneity is important. Spatial resolution is limited by the diffraction limit of light, and has not been tested to date in laser spot scanning studies, but remains a fertile area for further study.

#### ACKNOWLEDGMENT

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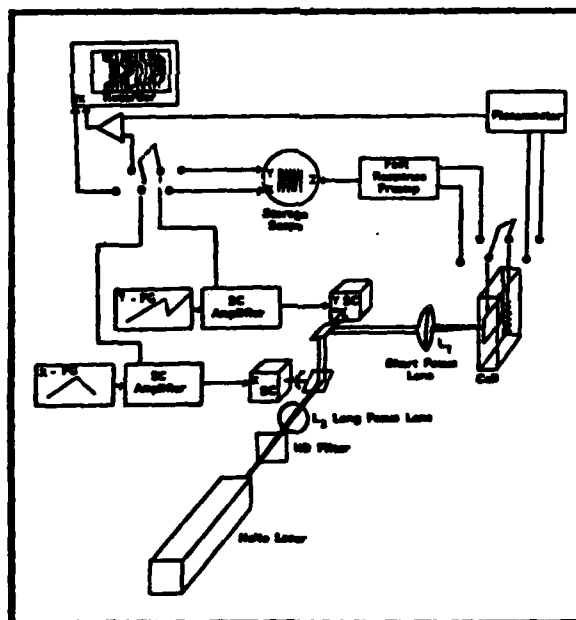
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**TABLE 1**  
**PEC PARAMETERS AS A FUNCTION OF SURFACE**  
**TREATMENT OF A CdSe FILM**

<u>Treatment</u>	<u>Photovoltage</u> (volts)	<u>J<sub>sc</sub></u> (mA/cm <sup>2</sup> )	<u>P<sub>max</sub></u> (mW/cm <sup>2</sup> )	<u>η</u> (%)
A - Annealing	-0.39	2.45	0.25	0.7
B - HCl Etch	-0.45	12.5	2.1	4.6
C - Anodization	-0.43	7.3	1.4	1.0

\*W-I lamp KG-2 filter, 45 mW/cm<sup>2</sup>  
Electrolyte: 1M Na<sub>2</sub>S, 1M S, 1M NaOH.



**Fig. 1. Pictorial representation of laser scanner.**

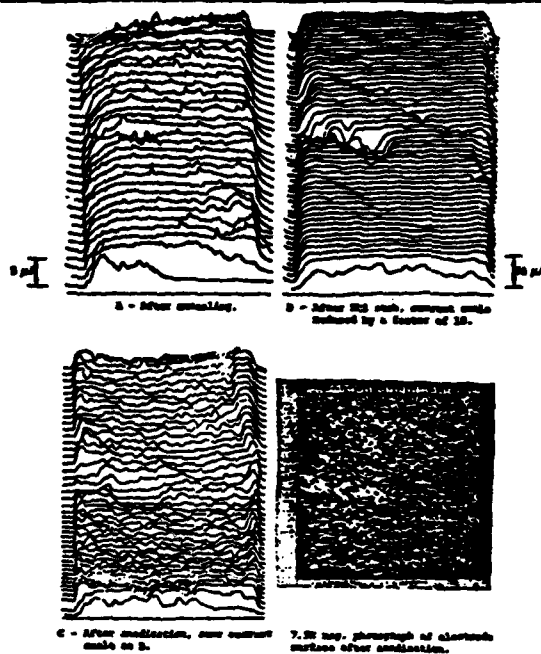


Fig. 2. He-Ne laser scan maps (623.8 nm) of CdSe thin film after A, annealing; B, HCl etch; C, anodization. Beam diameter  $\sim 20\mu$ , beam power at electrode surface  $\sim 100\text{ }\mu\text{W}$ .

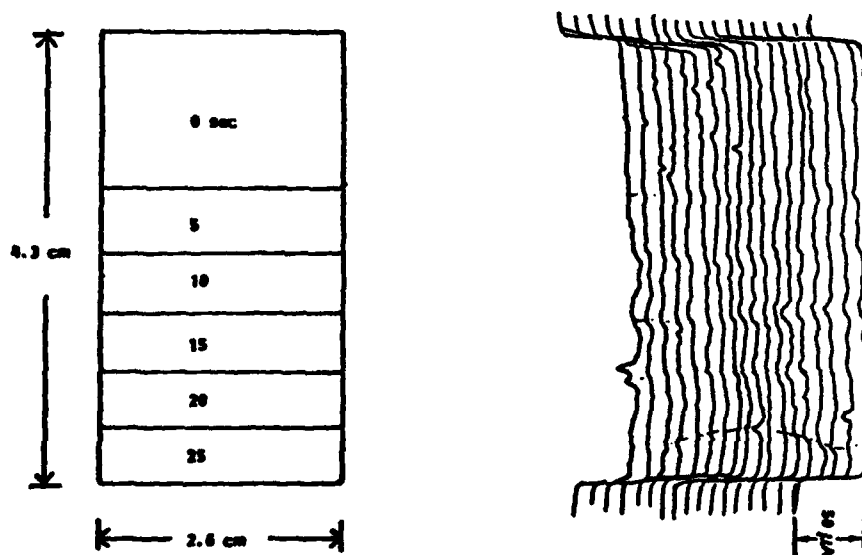


Fig. 3. Effect of etch time in 9M HCl on chemically deposited CdSe thin film. Cell biased at  $-0.4\text{V}$ . Diagram at the left indicates etch times for scan map on right. See Fig. 2 for laser parameters.

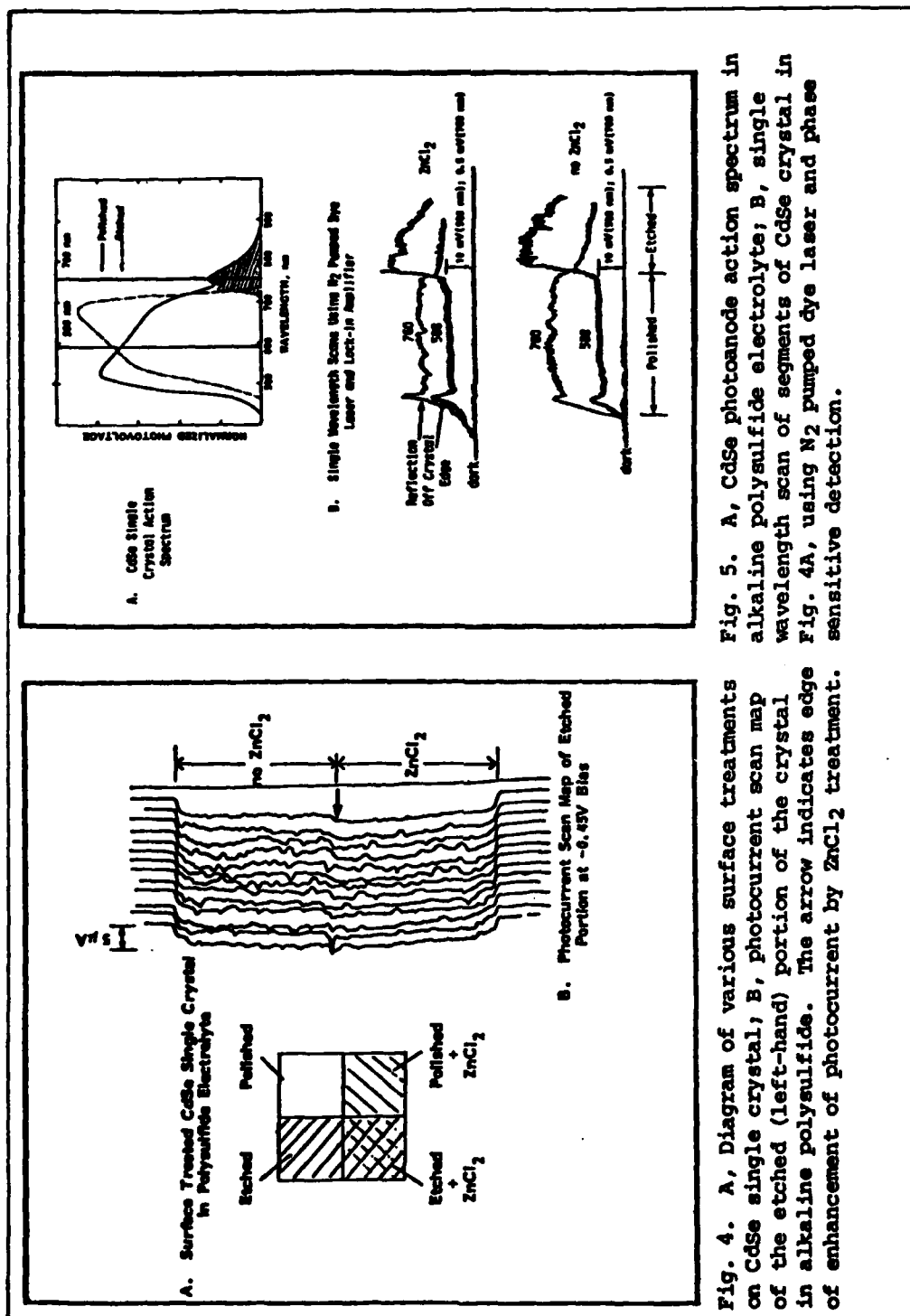


Fig. 4. A, Diagram of various surface treatments on CdSe single crystal; B, photocurrent scan map of the etched (left-hand) portion of the crystal in alkaline polysulfide. The arrow indicates edge of enhancement of photocurrent by  $\text{ZnCl}_2$  treatment.

Fig. 5. A, CdSe photoanode action spectrum in alkaline polysulfide electrolyte; B, single wavelength scan of segments of CdSe crystal in Fig. 4A, using  $\text{N}_2$  pumped dye laser and phase sensitive detection.

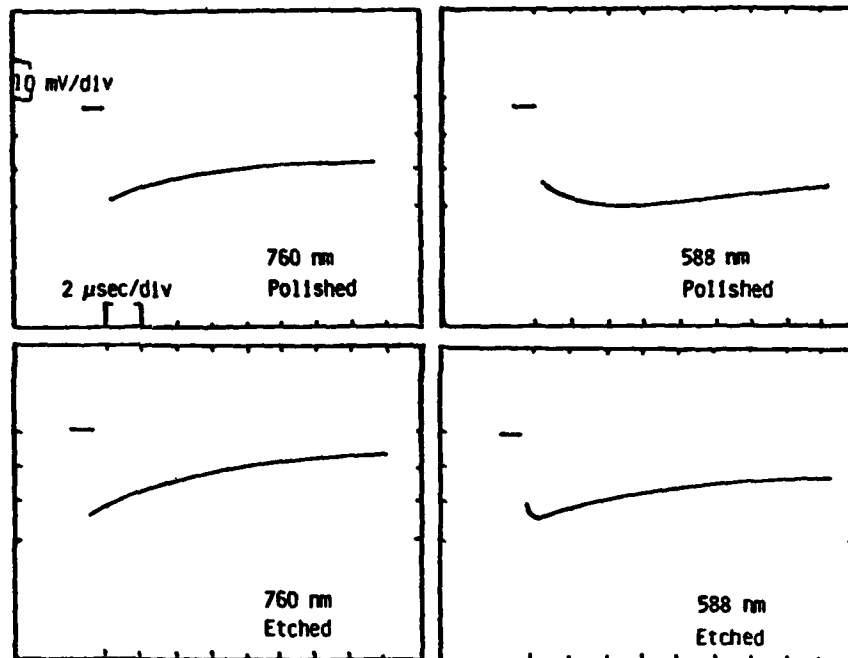


Fig. 6. Time resolved photovoltage of CdSe single crystal.

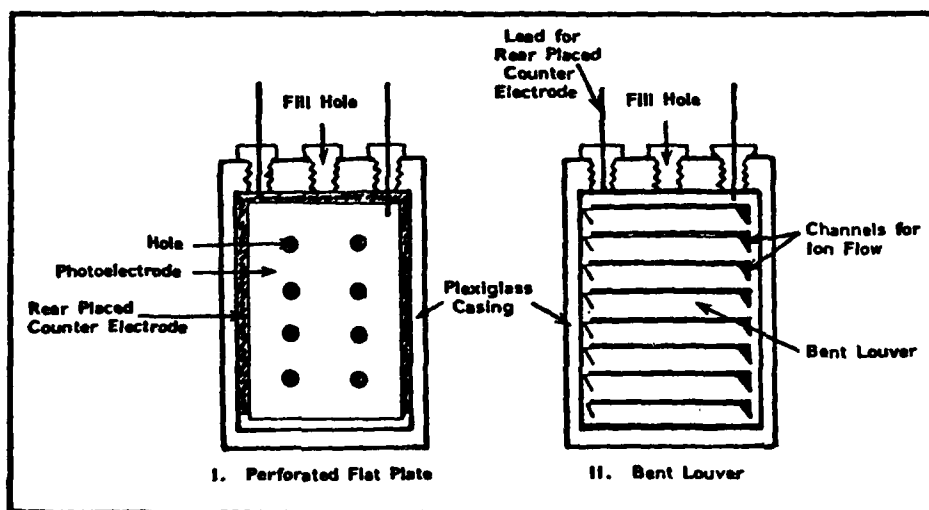


Fig. 7. Prototype frontwall thin film photoelectrochemical cells.

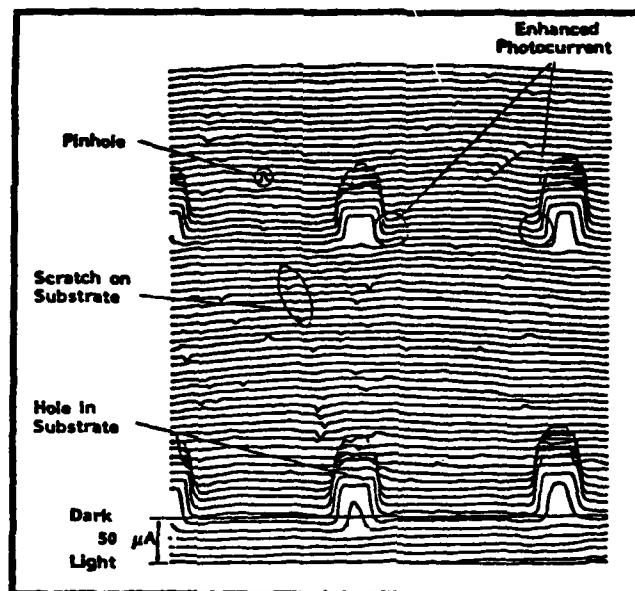


Fig. 8. Short circuit photocurrent scan map of chemical bath deposited CdSe film on a perforated substrate. Beam diameter  $\sim 50\mu$ ; laser intensity  $\sim 0.5$  mW.

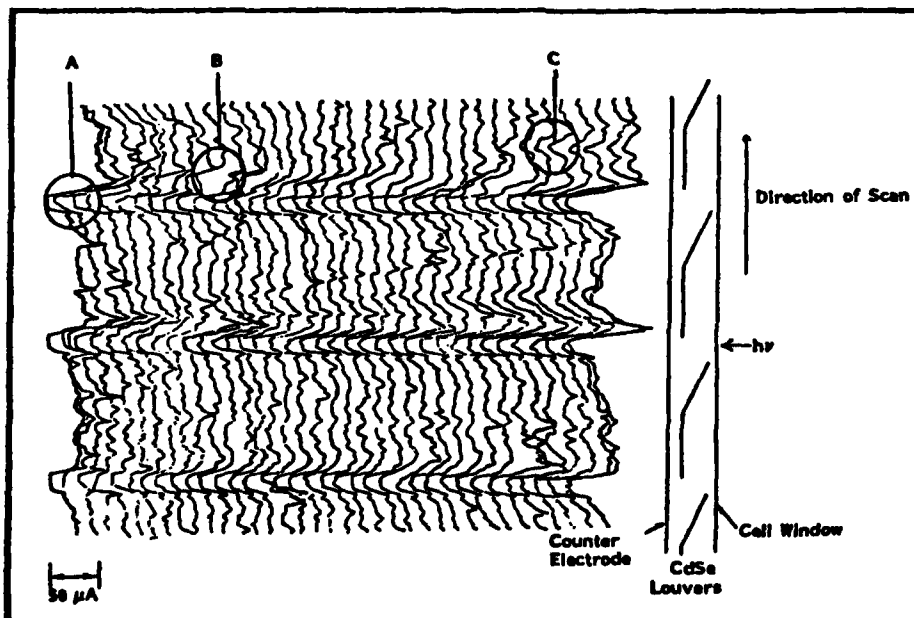


Fig. 9. Short circuit photocurrent scan of louvered cell; white light bias. Potentiostatted at 0.0V; chopped HeNe laser; lock-in amplifier. Electrodeposited film.

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